

PROJECT TITLE : ANALYTICAL INVESTIGATIONS
PERIOD COVERED : AUGUST 16 - SEPTEMBER 28
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MONO-, DI- AND TRI- CARBOXYLIC ACIDS

The reaction of carboxylic acids with ethyl iodide to produce the ethyl ester derivative has been found to be complex and unreliable. The procedure has been abandoned. A known method (1) has been modified for application to stem or leaf extracts using GC². Recoveries and standard deviation for each acid are listed in Table 1. Figure 1 illustrates the separation of acids found in a non-spiked tobacco.

GC²/MS HARDWARE MODIFICATION

A Carlo Erba static headspace injector (Mod. HS 250) has been modified and installed on the existing GC - MS (HP 5992). Experimental work will begin soon. A new injection block (Mod. HP 18740) for capillary GC has been installed.

REFERENCES

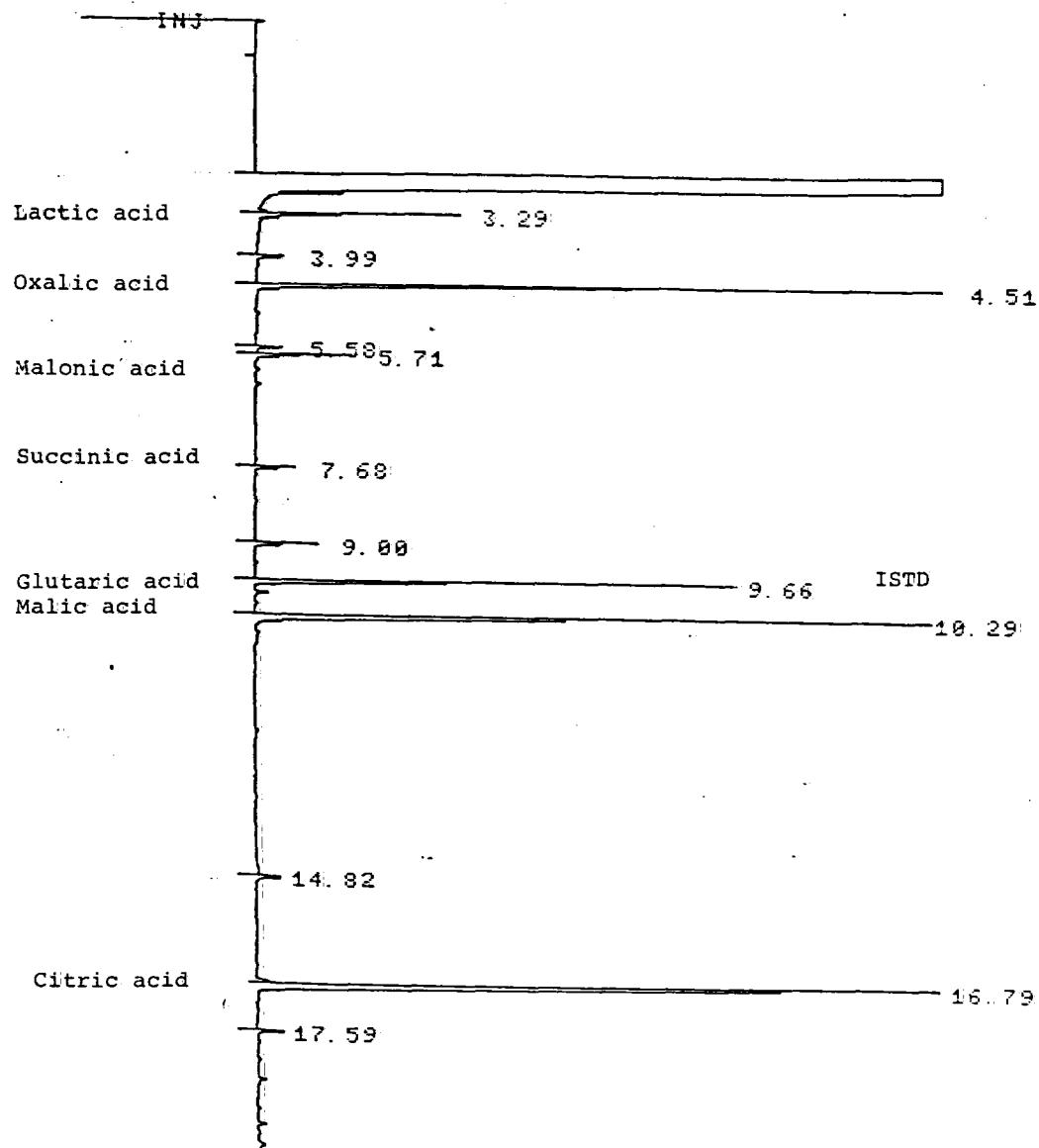
(1) Court-W.A., Journal of Chromatographic Science Vol. 16
p. 314 - 317, July 1978.

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ETL/jig/OCTOBER 2, 1981

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Figure 1 : Chromatogram of ethyl esters of mono-, di- and tri-carboxylic acids in non-spiked tobacco.
Stationary phase SP 2100; 50m x 0.32 mm i.d.



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(1) (1) (2)

	mg added	recovery %	%	% added	% found	recovery	CV 2.5
Lactic	10	106	-	0.32	0.32	100	
	30	100	-	1.58	1.57	100	
Oxalic	10	102	2.96	0.43	3.40	100	
	40	98		2.10	4.40	86*	8
Malonic	5	102	0.25	0.42	0.69	102	
	30	94		1.10	1.33	98	2.5
Pyruvic	5	101	-	0.41	0.48	117	
	50	98		1.03	1.21	117	
Succinic	10	103	0.04	0.21	0.22	88	
	30	98		0.52	0.56	100	
Fumaric	5	110	0.05	0.21	0.20	76	
	30	94	-	0.53	0.50	86	
Malic	10	100	3.60	0.40	3.90	97	
	60	97		2.00	5.10	91	5
Citric	10	97	4.80	0.55	5.20	97	
	40	88		1.10	5.41	91	8
				2.75	6.32	84*	

(1) Carried out with standards.

(2) On tobacco.

* Unrealistic results due to excessive addition of acids.

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